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HYDRAZINE PROCESS DEVELOPMENT

R. I. Miller
R. L. Pearson
F. R. Standerfer
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L. G. Carpenter
J. C. Whipple

AEROJET-GENERAL NUCLEONICS

Contract AF 33(600)-42996

Interim Technical Engineering Report
August through October 1963

The primary goal of this program is to develop, design, construct, and operate a continuous in-reactor hydrazine production loop based on the fissiochemical process approach.

This tenth quarterly progress report discusses operation of the hydrazine decontamination apparatus in the multicurie cell; the testing of ion exchange resins; the installation, testing, and modification of the AGN-302 Experiment at the MTR; liquid level probe difficulties; post-irradiation beamport plug removal and inspection; and analytical modifications.

Aeronautical Systems Division
Air Force Systems Command
United States Air Force
Wright-Patterson Air Force Base

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The hydrazine decontamination apparatus was installed in the multicurie cell at the CPP in Idaho; three mock runs disclosed several minor equipment and procedural deficiencies, which were corrected. Foaming in the distillation column reboiler was eliminated by removing metallic contamination from the feed ammonia. Several ion exchange resins were found to be effective in removing I-131 from hydrazine in both batch and column decontamination tests.

All remaining AGN-302 loop components were fabricated, shipped to the MTR, and installed; all systems were completely tested and modified, as required. Sequential testing, modification, and retesting required substantially more time than scheduled, delaying initial loop insertion until December. All equipment problems have now been resolved, except those caused by malfunction of the gas disengager liquid level probes. When repeated vendor and AGN modifications of the original circuitry did not significantly improve probe behavior, an entirely different approach to the exterior circuitry design and operation was initiated by AGN and, thus far, has allowed successful manual operation of the probes as liquid level controllers. A pneumatic serrated guillotine was purchased, modified, and tested for use in safely severing and sealing the beamport lines prior to removal of the plug from the reactor. The beamport plug will be withdrawn from the coffin in the TAN hot shop and transferred to the RML cell for dissection and examination. The Design Report and the Operating Manual were completed and distributed.

Hydrazine analytical procedures were revised to assure that no fission product iodine will be volatilized inadvertently. Sampling frequency and logistics have been assessed; sampler testing and operator training are in progress.

Loop operations at zero power, 1, 5, and 15 kw are now scheduled to begin on December 2, December 23, January 13, and February 3, respectively.

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FOREWORD

This Interim Technical Progress Report covers the work performed under Contract AF 33(600)-42996 from August through October 1963. It is published for technical information only and does not necessarily represent the recommendations, conclusions, or approval of the Air Force.

This contract with Aerojet-General Nucleonics, San Ramon, California, was initiated under ASD Project 7-840, Hydrazine Process Development. It was administered under the direction of Mr. Charles Tanis, Project Engineer in the Chemical Engineering Branch (ASRCTC), Manufacturing Technology Laboratory, Aeronautical Systems Division, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio.

Dr. John H. Cusack was the engineer in charge. Project engineers who cooperated in the developmental effort and in the preparation of the report were Dr. L. G. Carpenter, Mr. R. I. Miller, Dr. R. L. Pearson, Mr. F. R. Standerfer, J. C. Whipple, and H. J. Snyder.

The primary objective of the Air Force Manufacturing Technology Laboratory Program is to increase producibility, and improve the quality and efficiency of fabrication of aircraft, missiles, and components thereof. This report is being disseminated so that methods and equipment developed may be used through industry to reduce costs and give "MORE AIR FORCE PER DOLLAR."

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production program. Suggestions concerning additional Manufacturing Technology Laboratory development required on this or other subjects will be appreciated.

PUBLICATION REVIEW

AGN Report AN-1091

Reviewed by


J. H. Cusack
Program Manager

Approved by


D. E. Deutsch
Management Representative

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I. INTRODUCTION AND SUMMARY

A. BACKGROUND

This is the tenth Interim Technical Engineering Report on the Hydrazine Process Development Program (Air Force Contract AF 33(600)-42996) which is the second phase of a four-phase program leading to the production of hydrazine by fissiochemistry. The first phase (Contract AF 33(600)-40878) was completed in April 1961.

The primary goal of the current program is to develop, design, construct, and operate a continuous in-reactor loop, with the associated fuel handling and product purification sections. Direct support work necessary for the development of the loop and its components includes studies in decontamination, purification, fuel cycle, materials, chemical analysis, energy deposition, and reactor physics. Secondary support to the program includes capsule yield studies, basic studies of ion and free radical yield, and new product investigation. The results of these tasks will be coordinated to provide preliminary design and cost estimates for a self-critical pilot plant and prototype production plant.

B. PROGRESS DURING THIS QUARTER

Progress in each major area of the Hydrazine Program is discussed fully in later sections of this report; a brief summary is presented below.

1. Processing

The hydrazine decontamination apparatus was installed in the multicurie cell at the CPP in Idaho in such a way as to be easily removed and reinstalled. Initial testing, followed by three mock runs, disclosed several minor equipment and procedural deficiencies, all of which were corrected.

Previously observed foaming in the distillation column reboiler was traced to metallic contamination introduced with the feed ammonia; changes in vendor procedure eliminated this problem.

Several ion exchange resins, notably Dowex AG-1 x 10 (Cl^-), were found to be effective in removing I-131 from hydrazine in both batch and column decontamination tests. An ion exchange column may be used for final purification of the product concentrated and distilled from filtrate withdrawn from the AGN-302 experimental loop.

2. Materials

Fully enriched particulate UO_2 fuel was prepared and is now available in Idaho for use in the AGN-302 Experiment.

3. In-Reactor Engineering

All remaining loop components were fabricated, shipped to the MTR, and installed. All systems were completely tested and modified, as required. Operational deficiencies included several leaking field welds, final fitting problems, vacuum pump inadequacies, a vendor error in the makeup pump controller, and incapacity of certain manual bellows-sealed valves to seal across the seats under helium gas pressure. Because sequential testing, modification, and retesting required substantially more time than allocated in the schedule, initial loop insertion has been delayed until December.

By the end of October all problems had been resolved, except those created by malfunction of the gas disengager liquid level probes. These probes, as supplied with vendor instrumentation, proved incapable of reproducibly determining ammonia liquid level, although this capability was originally assured by the vendor. When repeated vendor and AGN attempts at modification of the original circuitry did not significantly improve probe performance, AGN initiated an entirely different approach to exterior circuitry design and operation which, thus far, has allowed satisfactory manual control of the probes to regulate liquid level. Automation of this system probably will not be completed before currently scheduled loop insertion; therefore, the liquid level probes will be manually controlled during preliminary loop operation.

A pneumatic serrated guillotine has been purchased, adapted, and tested for use in safely severing and sealing the beamport lines prior to post-irradiation removal of the loop from the reactor. After the lines have been severed, the experimental plug will be withdrawn from the HT-2 beamport to the removal coffin, and the entire assembly will be shipped to the Test Area North (TAN) facility. The plug will be removed from the coffin in the hot shop and transferred to the 10 by 35 ft radiation materials laboratory (RML) cell for dissection and examination.

The Design Report and the Operating Manual were completed and distributed; the Operating Manual will be continuously revised to reflect accumulated operating experience.

4. Support

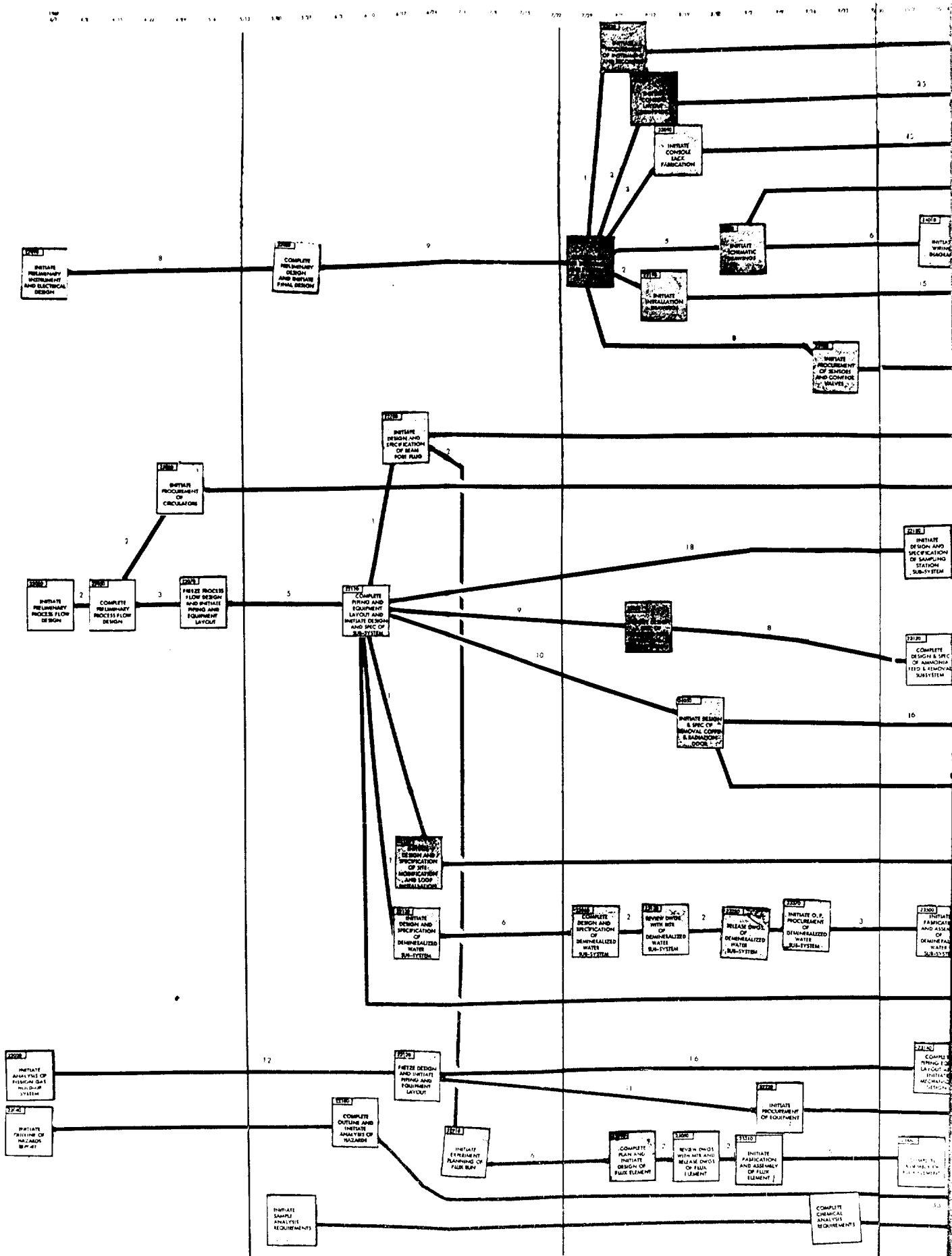
The hydrazine analytical procedure was revised to assure that no fission product iodine will be lost through volatilization.

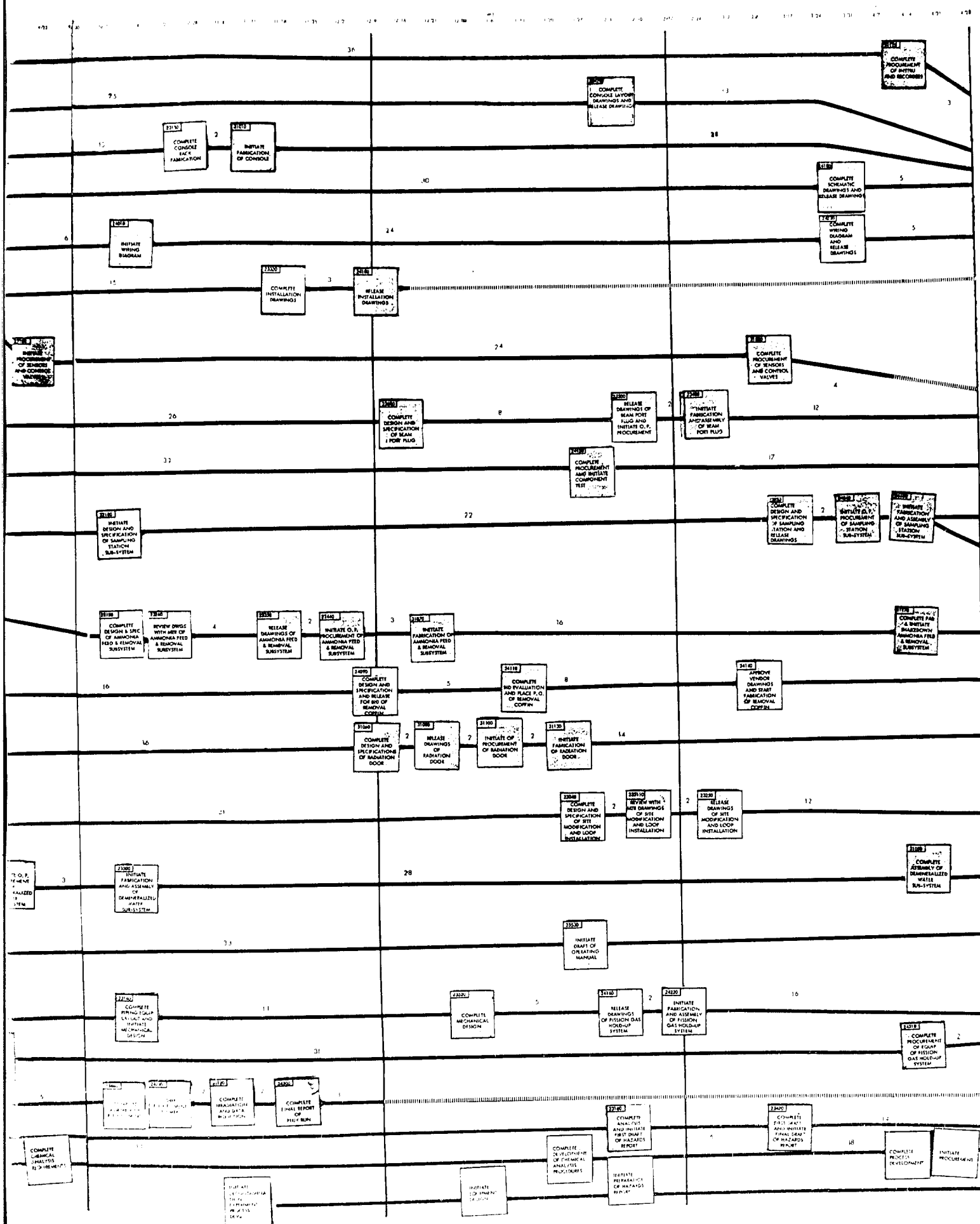
Development of chromatographic techniques for gas analyses continues in an effort to improve ammonia resolution.

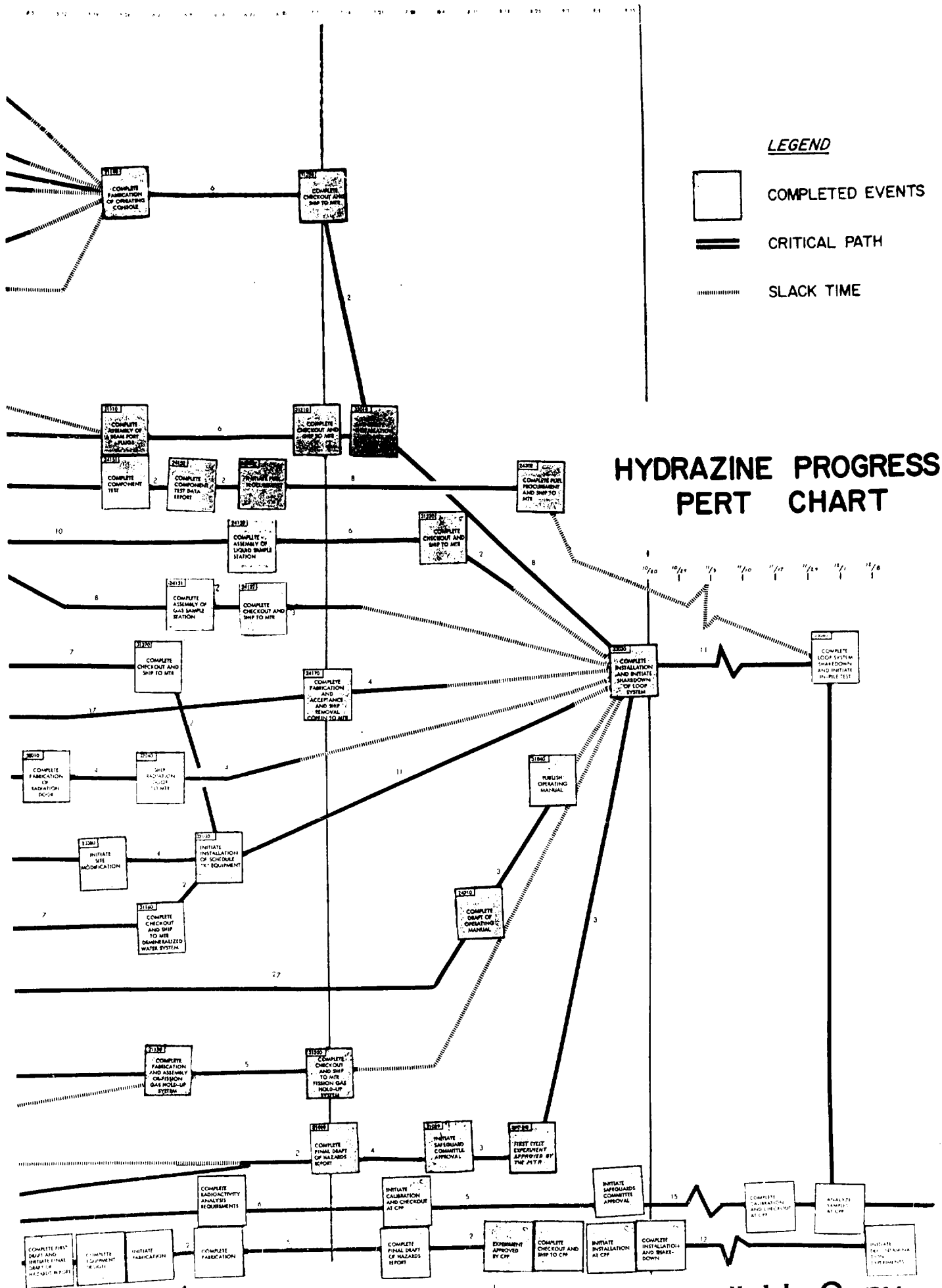
Sampling frequency and logistics have been assessed, the sampler tested, and operator training begun.

5. Scheduling

The PERT Network (Figure 1) illustrates the completion of essentially all tasks prior to loop insertion. Final shakedown and modification requirements beyond those initially contemplated have delayed loop insertion to December 2. Loop operations at zero power, 1 kw, 5 kw, and 15 kw are rescheduled for December 2, December 23, January 13, and February 3, respectively.







II. PROCESSING

A. EXPERIMENTS WITH DECONTAMINATION TEST MATERIALS

1. Compatibility Tests With Hydrazine

Several ion exchange resins and adsorbers were selected as candidate materials to further decontaminate the distilled hydrazine which will be withdrawn from the decontamination test apparatus. Samples of these materials were tested for compatibility with hydrazine by soaking them in hydrazine for three days (with periodic visual inspection), drying them to remove the hydrazine, and then recording their weights. Table 1 lists the resultant weight changes.

Activated charcoal has been rejected as a decontamination test material because of its vigorous reaction with hydrazine, even though this reaction may have been caused by oxygen adsorbed onto the charcoal. No other samples reacted significantly. Two other samples evolved noticeable heat when hydrazine was first added; materials to be used in a decontamination test will be pre-wetted with hydrazine to avoid any heat of adsorption during test.

TABLE 1
COMPATIBILITY OF HYDRAZINE AND CANDIDATE
DECONTAMINATION TEST MATERIALS

Test Material	Weight (mg)			
	Dry Sample	Hydrazine Added	Gross Change After 3-Day Exposure	Change in Sample After Re-drying
Ion Exchange Resins:				
AG-1-X10 Anion (OH ⁻)	1466	995	0	- 8
AG-1-X10 Anion (Cl ⁻)	1027	987	+ 2	- 9
AG-50W-X12 (H ⁺)	1032	878*	- 2	+106
Miscellaneous:				
Activated Charcoal	837	955*	-768**	- 51
Silver (precipitated)	1961	994*	+ 2	+ 1
Lead (granular)	2060	990	+ 3	+ 1

* Noticeable heat evolved during hydrazine addition

** Vial cover blew off during the first night

2. Batch Decontamination Tests Using Iodine-131

Two sets of batch tests were performed, using 1-gm samples pre-wetted with hydrazine (as described above). Samples of several other materials were included, but their compatibility with hydrazine was only qualitatively determined. Excess hydrazine was decanted from the samples and four milliliters of hydrazine containing approximately 1.5×10^7 dpm/ml carrier-free I-131 (as I^-) were added to the first set of samples; the same solution, containing approximately 7 gm/ml I^- carrier, was added to the second set of samples. Samples were agitated, allowed to settle, and a 200 λ aliquot of hydrazine was withdrawn for counting. Preliminary analysis of these data appear in Table 2.

TABLE 2
DECONTAMINATION OF I-131 IN HYDRAZINE
BY VARIOUS TEST MATERIALS

Test Material	<u>Adjusted Decontamination Factor</u>	
	Carrier-Free	7 gm/ml Carrier
3 controls (average)	1.0	1.0
Ion Exchange Resins:		
Amberlyst XN-1001	7.4	13.0
Amberlyst XN-1006	18.0	17.0
Dowex AG-1X10 (OH^-)	5.7	11.0
Dowex AG-1X10 (Cl^-)	10.0	20.0
Dowex AG-50W-X12(H^+)	0.9	0.9
Miscellaneous:		
Activated Alumina	1.0	1.1
Precipitated Silver	1.0	1.0
Granulated Lead	0.7	1.0
Silica Gel	0.9	0.9
Benzene	1.0	1.0

The control samples gave consistently anomalous results: the final count rate was 1.13 times the initial count rate. Although this anomaly has not yet been explained, it is assumed to be systemic and the results have been adjusted for it by dividing all final count rates by 1.13. Decontamination factors (DF) were calculated by dividing the initial by the final count rate.

The two sets of tests bracket the iodide concentration expected from the AGN-302 loop filtrate. It is evident that the carrier-added set gives higher DF than the carrier-free set, and that ion exchange resins were the only materials which performed substantial decontamination. The fact that ion exchange resins work well in a hydrazine medium may be extremely important for the final decontamination step in a hydrazine plant.

3. Column Decontamination Tests Using Iodine-131

An experiment was conducted to separate I-131 from hydrazine by ion exchange. The ion exchange column, having a theoretical capacity of 10 mequivaleints or 1.3 gm I^- , was filled with 6 ml N_2H_4 and 3.5 gm Dowex AG 1 x 10 resin (Cl^- form) and dried at $110^\circ C$. The dried column was prepared for runs by loading it with 6 ml N_2H_4 , letting it stand for 10 minutes, and flushing it with 10 ml N_2H_4 . The finished bed was 5 cm long.

Three 6-ml portions of N_2H_4 containing I-131 and I^- carrier were passed through the column at 1.5 ml/min, followed by two 6-ml portions of N_2H_4 wash, each of which was collected separately. The I-131 concentration was 0.4 mc/ml; the I^- carrier concentration was 7 gm/ml. The total iodide ion added represented 0.01% of the column capacity. Two hundred lambda aliquots were withdrawn from each portion collected, pipetted onto KI crystals, dried, and counted.

The results of this experiment, summarized in Table 3, show that each addition of N_2H_4 to the column pushed through the preceding addition with no detectable mixing. Since the iodide ion remained bound to the resin, it could not contaminate the wash.

TABLE 3
DECONTAMINATION OF HYDRAZINE BY ION EXCHANGE

Addition No.	Content	Effluent Contamination Factor
CV-1	$N_2H_4 + I^-$	No detectable count
CV-2	$N_2H_4 + I^-$	215
CV-3	$N_2H_4 + I^-$	325
CV-4	N_2H_4 wash	365
CV-5	N_2H_4 wash	No detectable count

This experiment was conducted in air with a short column, and the hydrogen ion concentration was not controlled; while these conditions may affect the operation of the ion exchange column, the results showed the decontamination factors to be 10 to 20 times higher than those observed in the batch test. Both the batch ion exchange and an improved column will be tested, using N_2H_4 concentrated and distilled from filtrate withdrawn from the AGN-302 experimental loop.

B. DECONTAMINATION TEST APPARATUS

1. Installation

The hydrazine decontamination apparatus was shipped to CPP and installed in the multicurie cell so that the minimum number of tubing lines and electrical cables must be severed and rebuilt for removal and reinstallation of the distillation skid. Figure 2 shows the main tubing and cable trunk entering the east face of the multicurie cell through an open access plug, with all tubing connections just outside the cell. Thermocouple connections are made, using standard thermocouple plug-jacks, inside the cell. Figure 3 views the apparatus through the main operating window. The air-operated sampler and the ammonia scrubber, the only other components of the apparatus to be located inside the multicurie cell, are mounted on the south wall, as shown on the right in Figure 4.

2. Modifications

Several deficiencies in the hydrazine decontamination apparatus were noted during testing; they have been corrected.

1) Valves: All round valve handles inside the hot cell were replaced with bar handles for easier operation by the master-slave manipulators. Reach rods, equipped with indexing pins to hold them in proper position and to give the operator a view of that position, have been installed on V-320, Hydrazine Distillate, and V-321, Ammonia Vent.

2) Ammonia Distillate Samplers: The cold-trap samplers have been replaced by 400 ml Pyrex traps with Teflon plug stopcocks on the inlets and outlets. Three ammonia distillate samples will be taken by installing these new samplers, one at a time, on a side stream from the ammonia vent line. Samplers are connected to the vent line by ball-and-socket joints secured by Thomas clamps.

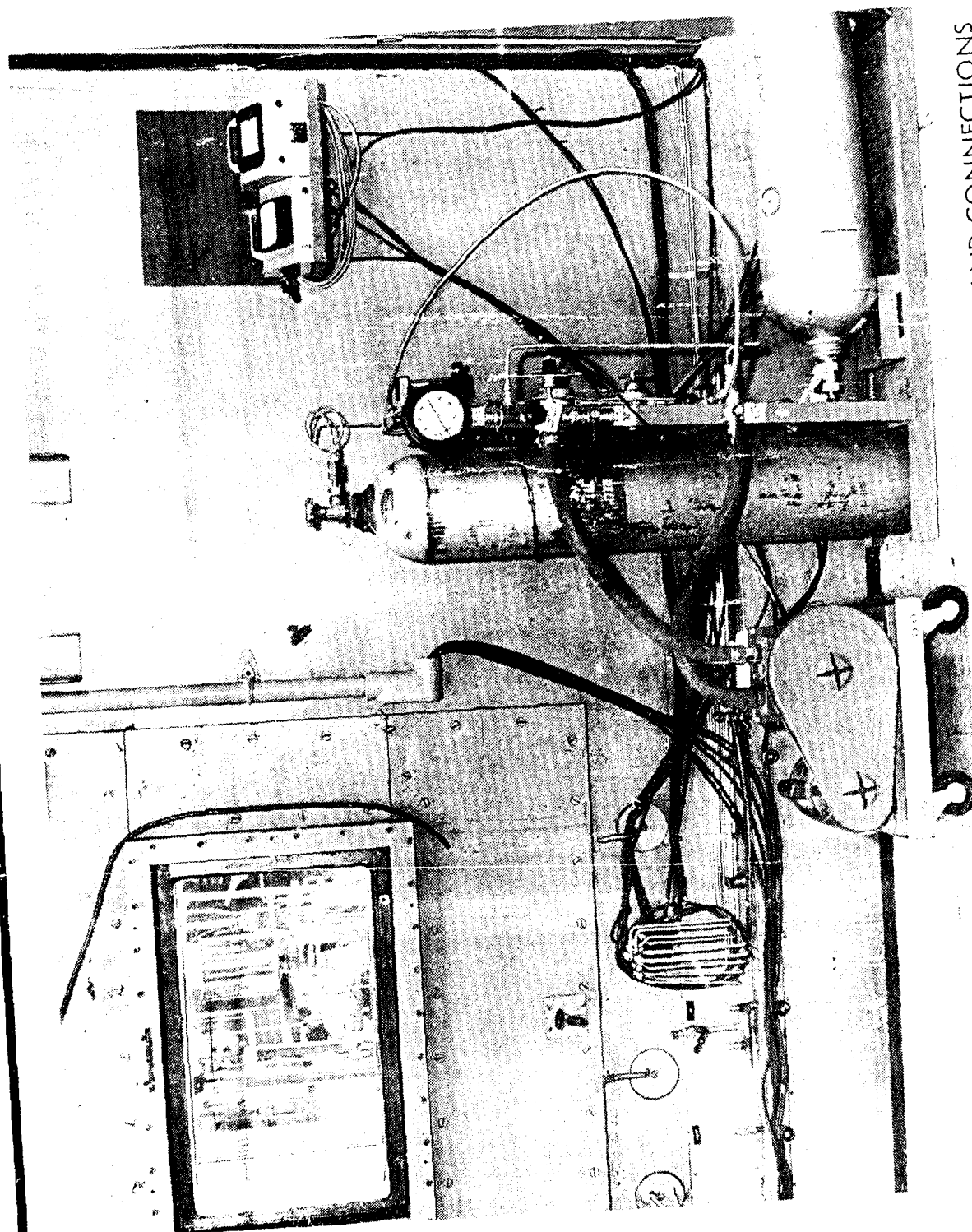


FIGURE 2. EAST FACE OF MULTICURIE CELL, SHOWING GAS SKID AND CONNECTIONS TO HYDRAZINE DECONTAMINATION APPARATUS

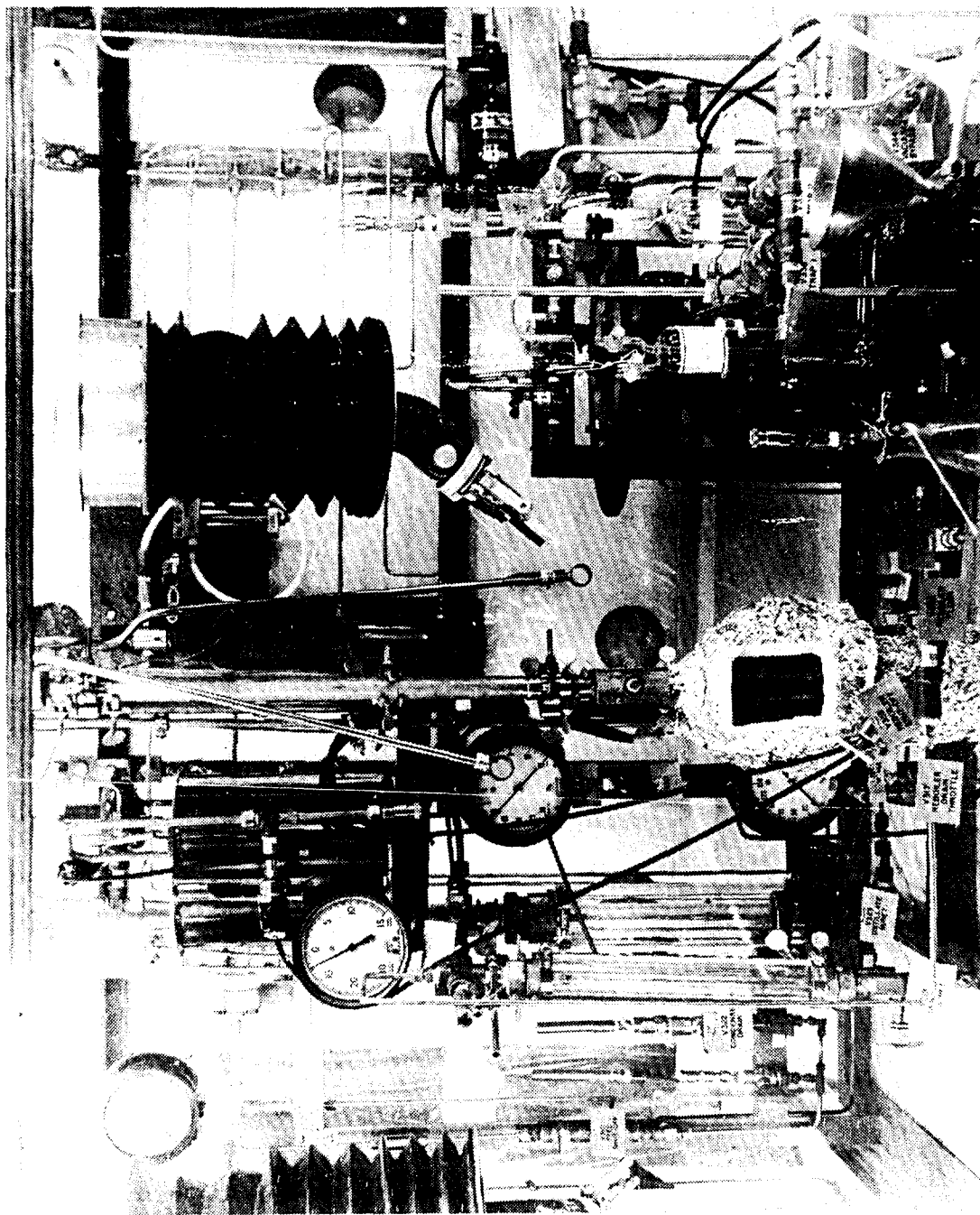


FIGURE 3. HYDRAZINE DECONTAMINATION APPARATUS VIEWED THROUGH MAIN OPERATING WINDOW OF MULTICURIE CELL

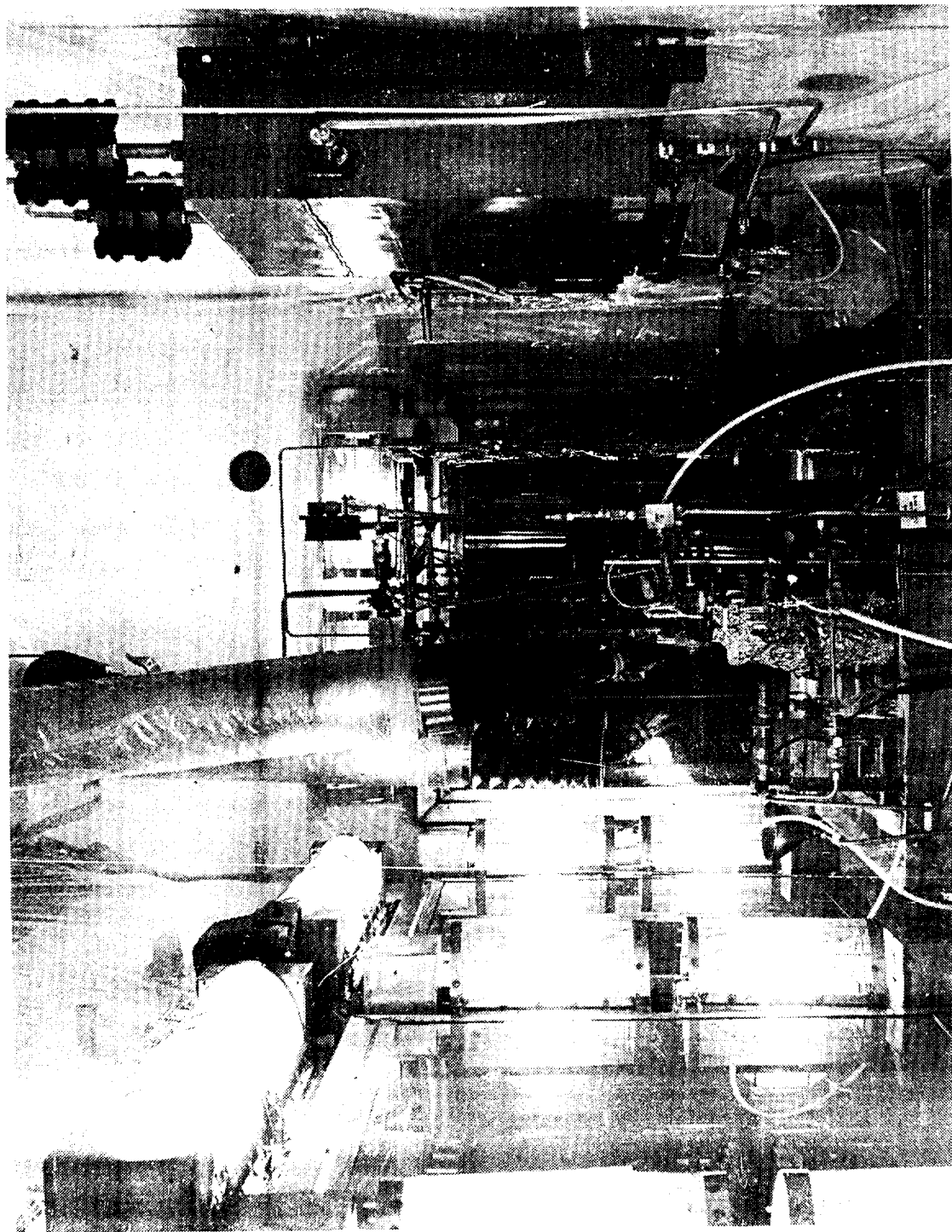


FIGURE 4. HYDRAZINE DECONTAMINATION APPARATUS VIEWED THROUGH OPEN DOOR
AT WEST END OF MULTICURIE CELL

For the initial tests, each sampler was preceded by a trap to prevent acid from being sucked back into the ammonia vent line; the tests have shown this precaution to be unnecessary.

Each acid trap is prepared for sampling by filling it with 200 ml of 6 N standardized sulfuric or acetic acid.

Ammonia vapors are forced through the sampler by the pressure drop (approximately 5 in. of water) across the ammonia scrubber.

- 3) Ammonia Scrubber: A circulating pump was added to the ammonia scrubber to afford maximum utilization of the citric acid neutralizing solution. The overflow outlet was lowered 1 in. to prevent citric acid from splashing into the exhaust bent.
- 4) Reboiler Drain Trap: A 500-ml Pyrex vacuum trap, enclosed in a steel beaker to serve as secondary container, was added as the reboiler drain trap. It collects the material (principally ammonia and dissolved contaminants) flushed from the reboiler between distillation runs, and evaporates the ammonia slowly through the hot vacuum hose to the ammonia scrubber.
- 5) Decontamination Units: The original decontamination units have been eliminated because the 1/4-in., quick-connect, Buna-N O-ring closed upon contact with liquid hydrazine, stopping the flow. The close dimensional tolerances of this connector lead to malfunction if the O-ring swells even slightly. One quick-connector set remains in each of the buret drains; the gaskets in both the body and stem have been removed, which will not affect the operation or safety of the hydrazine sampling procedure. Decontamination tests similar to the test described in Section A.2 above, will be performed in sample vials.
- 6) Pressure gage: A -30-in. water to 15 psig pressure gage has been added to the top of the hydrazine reboiler buret to indicate leakage.
- 7) Heater Power Shut-Off: The multipoint recorder has been incorporated into the heater power shut-off and alarm circuit. An open thermocouple, or a thermocouple indicating a temperature of 350°F, will trip the audible alarm and turn off the heaters until corrective action can be taken.

8) Feed Stop-Valve: A stop valve has been installed on the feed line between the Product Container connection and the feed line pressure gage. This valve is a 1/4-in. Hoke ball valve which can be closed from inside the cell by using the manipulators, or from outside the cell by pulling a cable attached to the valve handle.

9) Feed Sampler Mounting: The feed sampling station has been modified to include a sampler guide and a lever to facilitate engaging the quick-connectors during sampler installation.

The quick-connector stem assemblies on the feed line, which contain Buna-N O-rings, will be replaced with quick-connectors containing Teflon O-rings, as the Buna-N O-rings swell enough to close the quick-connectors (see above). Although the Teflon ring is not guaranteed by the manufacturer to seal tightly, it will seal well enough to stop rapid liquid flow.

10) Reboiler Sight Glass: The reboiler Pyrex sight glass was replaced with a clear glass of pure fused silica, more resistant than Pyrex to browning by radiation. With the spotlight installed to project light into the reboiler, the liquid level can now be observed very clearly through the operating window.

11) Product Container Connection: The Autoclave Engineers AE cone connection from the feed line to the Product Container is made by aligning the Product Container on the Distillation Skid, pulling the feed line into place, and engaging the gland threads with the valve body threads. The axial hole in the gland was enlarged to reduce alignment problems, and a tool was constructed for pulling the feed line securely into place.

Most of these modifications have already been tested; those which have just been completed will be tested during the next mock run.

3. Mock Runs

Three mock runs, using ammonia-5% hydrazine in a Product Container as feed, have been performed in the multicurie cell: The principal purposes of the runs were 1) to familiarize personnel with the operation of the apparatus, 2) to test the adequacy of the Operating Manual, and 3) to test the apparatus for remote operation.

Several minor deficiencies in the apparatus and Operating Manual became apparent during Mock Runs 1 and 2; these were corrected. All parts of the apparatus functioned satisfactorily during Mock Run 3, except the scrubber pump, which plugged; a procedural change will help prevent a recurrence. The check-out procedure was found to be unnecessarily long and is under revision. A tool is being fabricated to facilitate connecting the Product Container, and tools and jigs are being constructed to aid in connecting and handling the ammonia distillate samplers.

4. Reboiler Foaming

Foaming of the ammonia or ammonia-hydrazine solution was observed in the reboiler during all but two distillation runs at AGN. This foaming reduced the capacity of the distillation column and occasionally caused flooding. The cause of the foaming is suspected to be metallic contamination (principally copper and zinc) originating in the shipping cylinder. After the distillation column was thoroughly flushed with ammonia, 6 kg of liquid ammonia from the shipping cylinder were distilled, and foaming seriously reduced the column capacity. The column was again flushed thoroughly; the first flush yielded about 1 gm of brown solids shown by X-ray spectroscopy to be mainly zinc and copper. When 6 kg of ammonia were removed from the shipping bottle as vapor, condensed, and fed to the column to be distilled, no foaming was observed and the column did not flood. The first flush after this distillation yielded an estimated 0.25 gm of solids.

To assure delivery of a pure product, ammonia vendors thoroughly cleaned the shipping cylinders, filled them with high purity (refrigeration grade) ammonia, and then analyzed their contents for non-volatile solids. Spectrographic analysis of the ammonia has shown that metallic contamination is considerably below the 0.02% maximum specified for the product. This ammonia was used in Mock Run 3, and no foaming occurred.

C. HAZARDS REVIEW

The Hazards Report and the Operating Manual were published and distributed in September. The CPP Safeguards Committee has reviewed the Hydrazine Decontamination Experiment and granted preliminary approval to the experiment based on these two reports.

III. MATERIALS

One kg of 0.7 to 1.0 μ particles of 93%-enriched UO_2 was received and assayed as 93.074 wt% U-235 and 87.67% uranium. Permission was received from the AEC to process the fuel by firing it to 1300°C for four hours, cooling, crushing, and screening it through 100-mesh screens. The processing of the fuel was concluded in September, and all 1000 gm were shipped to NRTS for storage until required in the AGN-302 loop.

IV. IN-REACTOR ENGINEERING

A. LOOP DESIGN, FABRICATION, AND TESTING

All remaining AGN-302 loop components, including the valve box shields, were completed and shipped to the MTR. Spare parts retained at San Ramon were collected and shipped.

The permissible operating limits for the experiment were established (as tabulated below) on the basis of the design capacity of the demineralized water system, ammonia condenser, and slurry loop pressure relief system:

Fission power, kw	1	5	15
Nominal operating pressure, psi	275-1200	425-1200	950-1200
Gas disengager temperature, °F	106- 121	109- 130	127- 140

A study was made to determine the pressure rise in the slurry system, caused by fission product decay heating of trapped ammonia vapor, after the loop is removed from the reactor. Results show that a maximum pressure rise of 9.8 psi will occur if the loop is removed one day after shutdown, and 5.0 psi if the loop is removed five days after shutdown.

Lines and valves in the liquid sampling station were measured volumetrically, as was the external piping from the sampling station to the nitrogen pressurization bottle racks (after loop installation). These measurements were used to formulate a procedure for removing filtered ammonia trapped between valves during isolation of the 1-cc liquid samplers. The procedure involves pressurizing the external lines to blow the trapped ammonia back into the slurry loop; since no ammonia is flashed, soluble contaminants cannot deposit in the piping.

On completion of the AGN-320 experiment, all lines between the valve box and the experimental beamport plug must be severed and sealed before the plug can be removed from the reactor. The 1-in. pipes (containing only demineralized water) will be drained, sawed apart, and plugged. The 1/4-in. process lines will be crimped, clamped, and guillotined. A pneumatic, serrated guillotine was purchased for these operations. One set of teeth was ground down and shimmed for crimping, and the other set will be used for cutting. Clamps for use after crimping were designed and fabricated. An

enclosure was constructed which will be attached to the experimental plug to provide secondary containment for the crimped tubes.

The crimping procedure was checked out with a mockup of the process tubing bundle having the same dimensions and greater rigidity than the actual tubing to be used in the experiment. The seven tubes were crimped, clamped, and cut in a space small enough to fit inside the cut tube enclosure. During crimping and cutting, the tubes were pressurized to either 250 or 25 psig; in the latter case, the pressure was increased to 250 psig for leak-checking, since no leaks were detected at 25 psig. Helium leak rates of 10^{-4} to 10^{-9} cc/sec observed at the higher pressure correspond to less than 1 mg/hr of ammonia; since the maximum pressure inside the loop should be 10 psig, the clamping procedure is considered satisfactory. Figure 5 shows the guillotine in place for crimping the first tube in the mocked-up bundle. Figure 6 shows six of the seven tubes cut and clamped, with the cut tube enclosure in the background.

After all lines have been severed, the experimental plug will be transferred from HT-2 to the removal coffin on a 76-in. tray. Close fits between the plug and HT-2, and between plug and coffin, necessitate precise alignment of the HT-2 and coffin centerlines. An alignment tool to locate the centerline of the 22-in. part of HT-2 and project it out through the coffin position, was designed, fabricated, and shipped to the MTR. It consists of a precision-ground flat plate resting on the bottom of HT-2; a post at each end locates the center of the hole, and this centerline is projected out through the coffin by means of a length of piano wire.

Since the as-built capacity of the product removal vessels was measured to be 7.787 liters, or less than the design value of 8.5 liters, the nitrogen pre-pressurization value for a 5-liter sample had to be recalculated. This calculation was performed for various slurry loop operating pressures between 600 and 1200 psig; the result is a linear function varying from 79 to 274 psig.

The slurry loading bomb was modified to allow nitrogen and ammonia to enter it from the bottom, which, AGN tests have demonstrated, will prevent plugging of the bomb outlet.

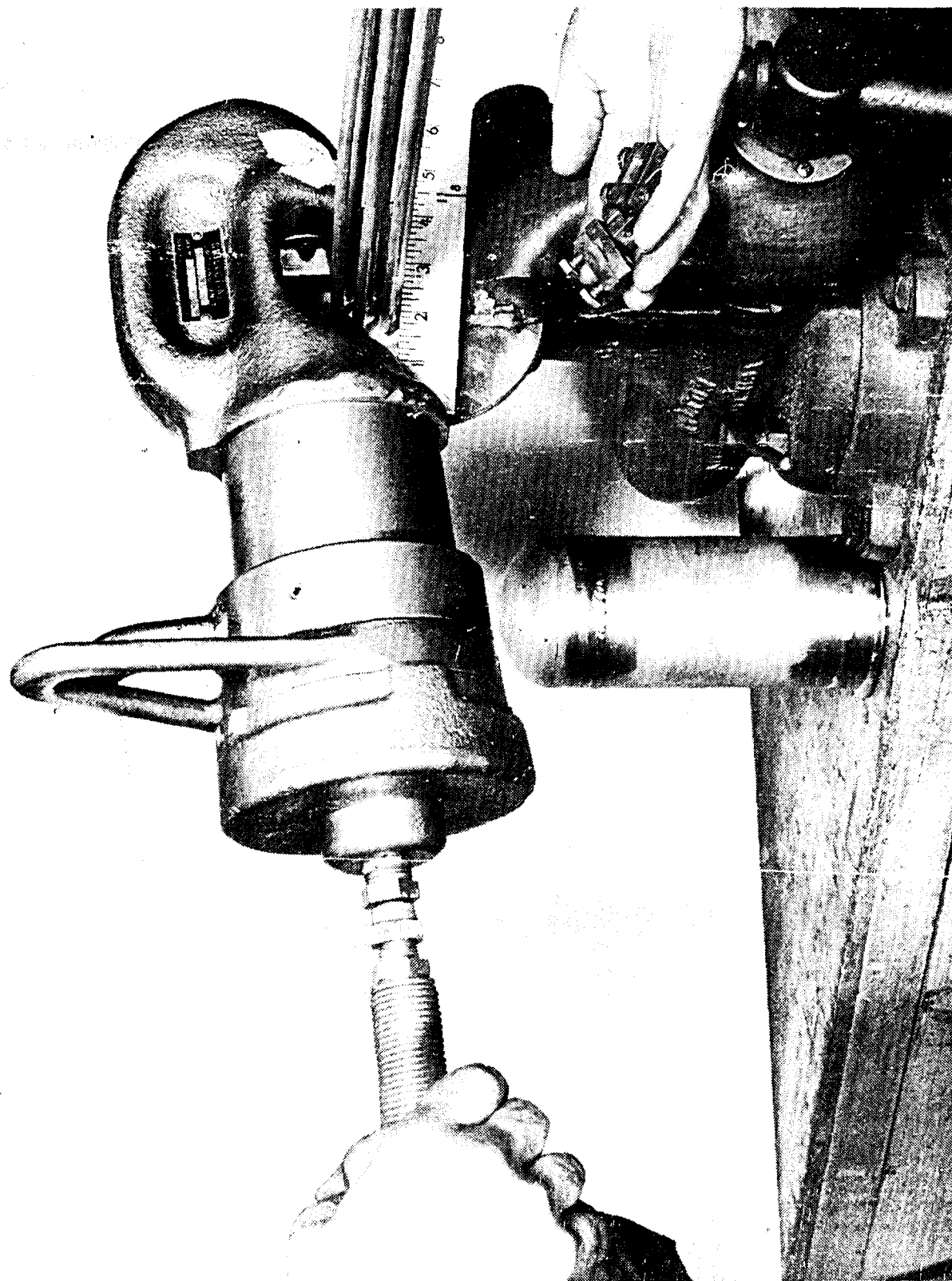


FIGURE 5. GUILLOTINE IN PLACE FOR CRIMPING FIRST TUBE IN MOCKED-UP BUNDLE

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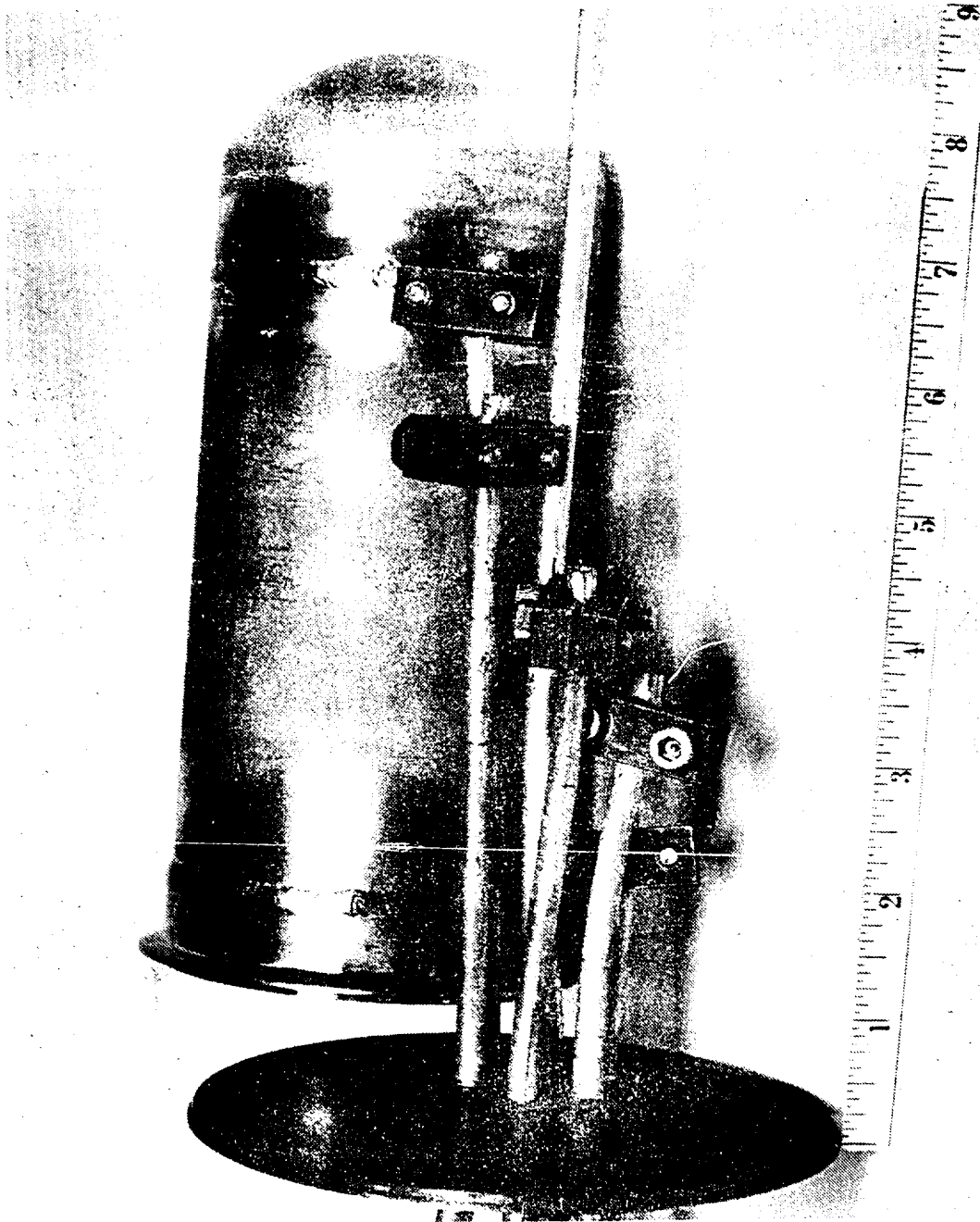


FIGURE 6. MOCKUP OF PROCESS TUBING, SHOWING CUT TUBE ENCLOSURE IN BACKGROUND

Connections were added to the gas and liquid sampling stations so that nitrogen pressurization and Freon-12 leak-testing can proceed simultaneously, precluding air contamination of the system while the nitrogen and Freon-12 bottles are interchanged. Drawings were revised, and the MTR sampling station piping altered accordingly.

B. INSTRUMENTATION AND CONTROLS

Various modification procedures were written and transmitted to PPCo to facilitate loop installation.

The 0 to 5% hydrogen analyzer, found to be defective in September, was repaired at the vendor's plant and reinstalled; a replacement was purchased and delivered to the MTR.

Replacements for the superheater power control coils were procured; an alternate power control system was assembled at AGN and shipped to the MTR.

An extension plate for the gas analyzer panel was manufactured and shipped to NRTS for installation.

To eliminate the high electrical noise level of the existing makeup pump pacer control, a replacement electronic timer was designed and is being fabricated; it will be mounted on the Control Console.

The annunciator was redesigned to prevent spurious noise signals from tripping unactuated annunciator components.

Vacuum gages for the gas and liquid sampling stations, a pressure gage for the liquid sampling station, and a defective power supply within the loop pressure signal conditioner were replaced, installed, and satisfactorily tested.

Action was taken to correct the erratic behavior of the gas disengager liquid level sensor: New trip circuits were designed to supplant the unstable circuits in the vendor-supplied, 20-kc, gas disengager liquid level controllers. Persistent erratic behavior suggested that the oscillator portion of the controller was also faulty. It was then determined from tests on the probes alone that performance would be improved by operating at 78 kc, instead of in the 20-kc region for which the controllers were designed, and that the wet-to-dry transition could be detected by attaching a commercial

oscillator to each probe and monitoring the probe output signal on an oscilloscope. Accordingly, four oscillators were procured and delivered for installation in the beamport area.

A manual switching system to permit oscillators and readout equipment to be located at the Control Console was designed and components for it were ordered. Parallel investigations were initiated to procure an automatic switching system for later implementation in the AGN-302 loop.

C. LOOP INSTALLATION AT THE MTR

During August, the outside contractor completed his work in the Annex Enclosure, and as-built construction drawings were transmitted to PPCo; PPCo recalibrated the process instrumentation and installed cables, cable trays, and interconnecting electrical wiring.

In the beamport area, the valve box, liquid sampling station, experimental plug, bottle racks, and associated process piping and double containment lines were installed. Product removal and liquid sampling casks, with their piping, were lodged next to the liquid sampling station (Figures 7 and 8). All the doubly contained process piping, surrounded by lead shielding segments, was installed in the MTR basement in August.

The loading of the experimental plug onto the insertion tray and then into the removal coffin was successfully tested. This procedure will be repeated before startup of the in-pile run, when the plug will be loaded into the beamport hole, backed into the coffin, and then reinserted into HT-2.

Pressure testing of the ammonia and slurry systems began in early September. The several leaking welds discovered in the ammonia pit and liquid sampling station were repaired. Equipment checkout revealed 32 minor malfunctions, most of which were repaired by September 16. Subsequently, the manual, bellows-sealed valves in the sampling stations were found to leak across the seats under helium gas pressure. Since PPCo stipulated the requirement of helium leak-tightness for both liquid and gaseous systems, 27 valves were returned to the vendor for replacement of stainless steel bonnet tips with high density polyethylene, and reinstalled in the sampling stations the first week in October. Defective vacuum gages in the sampling stations (damaged during pressure testing by the valve seat leakage) were replaced, as was a pressure gage in the liquid sampling station.

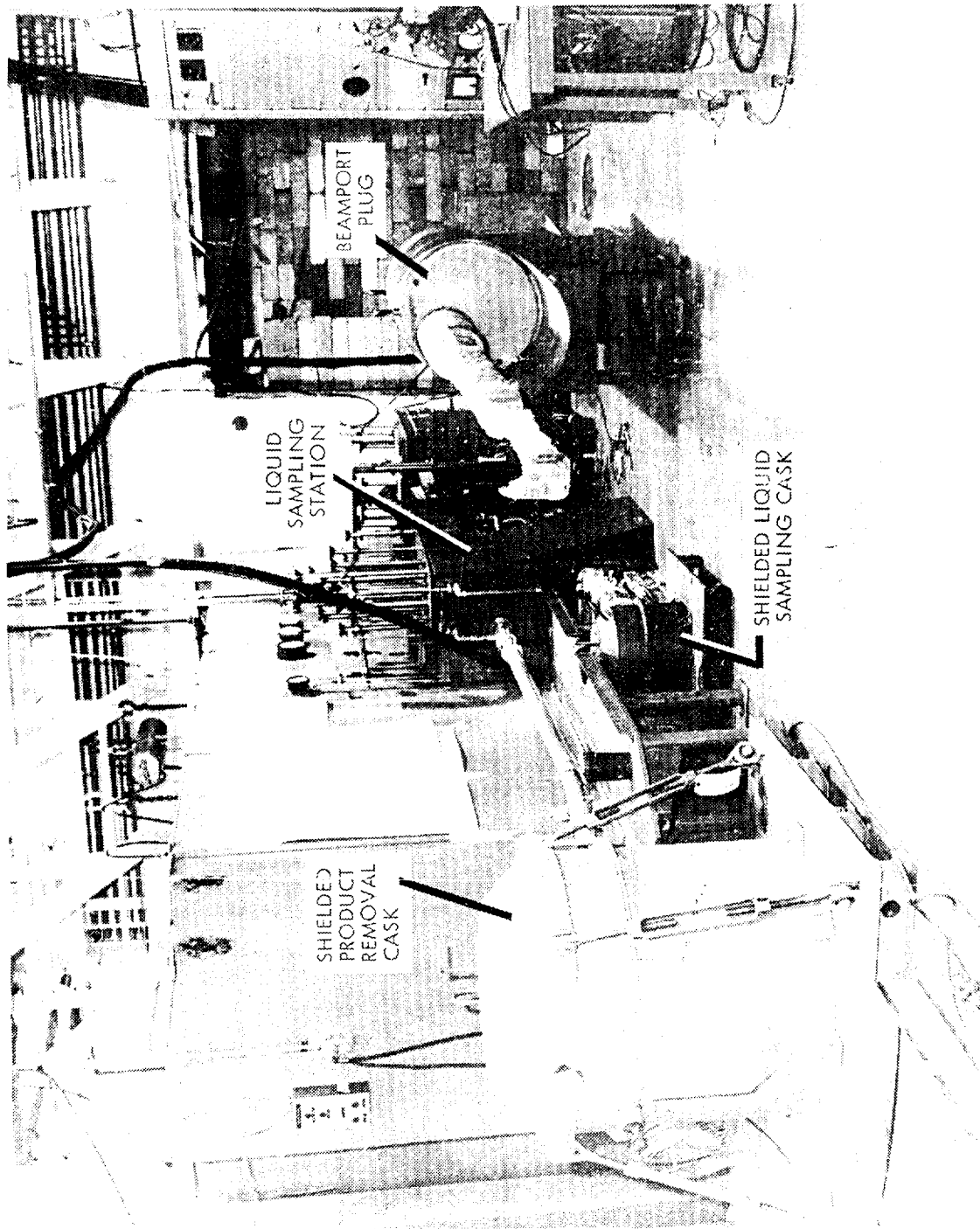


FIGURE 7. PRODUCT REMOVAL AND LIQUID SAMPLING CASKS

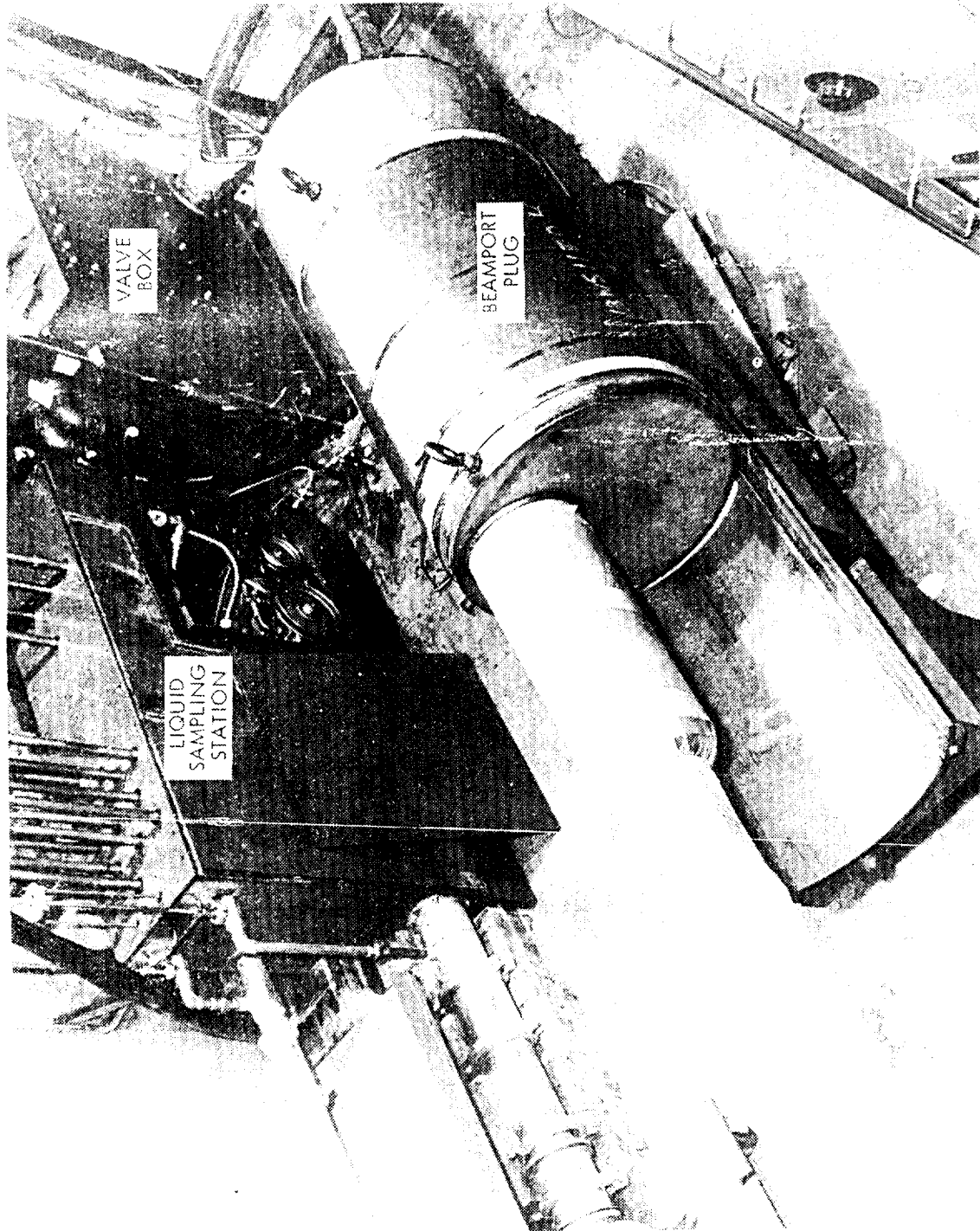


FIGURE 8. LIQUID SAMPLING STATION

These repairs and substitutions delayed introduction of ammonia into the ammonia feed and removal system and the slurry system until October 6, when the ammonia makeup pump, which had functioned satisfactorily under atmospheric conditions at San Ramon, was found to be operating at only 1/10 its specified value. After various trouble-shooting procedures had failed, the vendor's chief engineer, dispatched to the MTR, disclosed that the wrong stroke frequency control pacer had been furnished with the pump. After the pacer control had been modified on site to promote adequate delivery, the slurry loop was repeatedly filled with ammonia and the slurry circulator was operationally tested. Since the gas disengager liquid level system continued to behave erratically, the following corrective steps were taken:

- 1) The makeup pump pacer control unit, which had been producing electrical noise spikes in the liquid level control circuitry, was physically relocated to the Annex Enclosure.
- 2) New, magnetic amplifier trip circuits were fabricated at AGN and installed at the MTR, replacing the limited-range vendor circuits. This approach, based on the assumption that the oscillator portion of the vendor-supplied equipment was adequate, but did not yield performance improvement.
- 3) The vendor twice dispatched specialists to the MTR to make test measurements and to attempt to improve the output signals with new control boxes.
- 4) Results of tests on the probes in both the liquid and dry states indicated that sympathetic vibrations were creating a cross-coupling effect between probes, and that, because of the probe face-to-inner wall spacing, each probe was interfering with itself through acoustical standing wave reflections. It was also determined that the probe output signals were lower than expected, and that the dry-to-wet signal ratio was about 2:1 instead of the desired 10:1.
- 5) The external control units supplied by the vendor were removed from the equipment because they proved inadequate for the relatively low probe output signal and the small dry-to-wet signal ratios.

6) During the probe tests referred to above, it was noted that, if a probe were wetted after an oscillator had been connected to it and adjusted in frequency until a probe output signal peak was attained, the signal would decrease by a factor of 2 to 3, and furthermore, that each probe peak frequency and output signal differs from that of every other probe. Accordingly, four oscillators were procured for probe excitation, and an oscilloscope will be used to read the peak output voltages.

D. POST-IRRADIATION EXAMINATION PLANNING

Post-irradiation examination of the experimental beamport plug was discussed with PPCo Metallurgy and Hot Cell Branch personnel. AGN representatives toured the Test Area North (TAN) facility, paying particular attention to the large hot shop, the 10 by 35 ft radiation materials laboratory (RML) cell, and the smaller general purpose cells. The experimental beamport plug will be delivered from the reactor to the hot shop in the removal coffin, withdrawn from the coffin, and transferred to the RML cell (adjacent to the hot shop) for dissection and examination. Insertion of the plug into the cell will involve loading the plug onto a shelf attached to the RML cell side of the door and rotating the door closed. Because of weight limitations on the shelf, the lead shield will probably have to be removed in the hot shop. The dissected plug components will be buried in concrete containers instead of in the removal coffin so that the coffin can be reused.

E. REPORTS

The Hazards Report (AN-TM-186) was expanded to include material on slurry loop pressure and temperature operating conditions and on the effect of MTR power reduction rates. Sections on the fuel induction system were revised to incorporate design changes in slurry loading components. AGN personnel assisted PPCo project engineers in preparing the Hazards Report for submittal to the PPCo Hazards Committee. Four copies of the Hazards Report were distributed to the AEC office in Idaho.

The Operating Manual was completed and distributed, and 20 copies were delivered to PPCo for use in operator training. Following sequential checks of Operating Manual procedures by AGN personnel in Idaho, three copies were revised for use in actual loop operation during the out-of-pile shakedown tests.

V. SUPPORT

A. SAMPLE SCHEDULING AND HANDLING

Desirable sampling frequencies have been discussed with MTR and CPP operations and the logistics of sampling, transport, analysis, decontamination, and return of sampling devices has been assessed. Sampling procedures were evaluated, and the equipment operationally tested for compatibility with the procedures. Operator training for sampling operations is in progress. Sample shipment by a single truck having a capacity of 10 sample casks was requested; a review of sampling frequencies and equipment available indicates that this is feasible.

Some of the gas and liquid samplers have been checked at CPP for volume calibrations, remote handling capability, and compatibility with analytical equipment. Two liquid sample reaction units were fabricated at CPP; both will be used in routine work unless activity levels require analyses to be performed in a hot cell.

B. ANALYTICAL METHODS

1. Liquid Sample Reaction

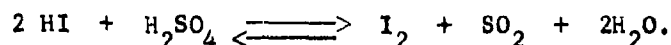
The use of sulfuric acid causes significant heat generation in the sample reaction step. It is known that iodide ion can easily disproportionate in hot acid solution to form H_2 and I_2 . If fission product iodide is present in appreciable concentration, some could easily be volatilized by this process.

As a precautionary measure, the analytical group at CPP suggested that acetic acid replace sulfuric acid for the sample reaction step. Experimental work at CPP has shown that iodine probably is not released when the sample is reacted into acetic acid. Also, acetic acid will not interfere with the chemical analysis methods to be used for ammonia and hydrazine. A qualitative test of the acidification of a $KI-NH_4OH$ solution, and a radiochemical analysis of $I^{131}-NH_4OH$ solutions before and after acidification, were performed at AGN to evaluate this substitution.

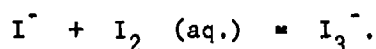
a. Acidification of $KI-NH_4OH$ Solutions

Three ml of concentrated NH_4OH (corresponding to 0.046 equivalents on the amount of NH_3 found in the 1.4-cc liquid samplers) were

put into 25-ml volumetric flasks to which a few mg of solid KI were added. To one of the flasks, 2 ml of concentrated H_2SO_4 were than added. The reaction was vigorous, and the resulting solution hot. The presence of I_2 was observed, and the characteristic odor of I_2 was detected. The reaction can be expressed by:



When the flask was stoppered and shaken, the color disappeared, and the resulting solution was colorless. Iodide solution readily dissolves iodine with the formation of triiodide:



To one of the remaining flasks, 3.2 ml of glacial acetic acid were added, producing a less vigorous reaction with no evidence of I_2 release.

b. Acidification of $\text{I}^{131}\text{-NH}_4\text{OH}$ Solutions

Three ml of concentrated NH_4OH were added to each of five, 25-ml volumetric flasks. One ml of an I^{131} solution was than added to each flask and the samples treated as tabulated below:

Flask No.	N_2H_4 Added	Acidification Step
I	None	None
II	None	3.2 ml glacial acetic acid
III	None	2 ml concentrated sulfuric acid
IV	None	2 ml concentrated sulfuric acid
V	0.7 mg	2 ml concentrated sulfuric acid

The samples reacted with H_2SO_4 , unlike those reacted with acetic acid, became hot enough to create steam. The flasks were unstoppered, the solutions allowed to cool for an hour and a half, and the samples diluted to volume with H_2O . Ten ml aliquots were then pipetted into 4-dram, glass, screw-cap vials and counted for one minute in a well chamber, using a gamma spectrometer initially checked with an I^{131} standard. The results from the counting study are tabulated below. (The counts are corrected for the background count of 49 cpm.)

<u>Flask No.</u>	<u>Acidification</u>	<u>Counts per Minute per 10 ml Aliquot</u>
I	None - Reference	3449
II	Acetic acid	3418
III	Sulfuric acid	3535
IV	Sulfuric acid	3473
V	Sulfuric acid	<u>3455</u>
Average of five samples		3466

Since the expected deviation of this count rate is 59 cpm, the samples (except that from Flask III) fall within the counting statistics. The standard deviation was calculated from all the results to be ± 43 cpm, giving a relative standard deviation of 1.24%. These data show no loss of I_2 upon acidification with H_2SO_4 .

When last counted (23 August 1963), the I^{131} solution gave a value of 5.015×10^5 dpm per ml. After a decay time of 34 days (these five samples counted 26 September 1963), the activity of the I^{131} solution was:

$$N = N_0 e^{-\lambda t} = 5.015 \times 10^5 e^{(-0.0856)(34)} = 2.51 \times 10^4 \text{ dpm per ml}$$

From the average activity of the five samples counted (3466 cpm per ml aliquot), the activity of the original I^{131} solution would be (3466) 25/10, or 8.665×10^3 cpm per ml of I^{131} solution. This count rate indicates a geometry factor for the well crystal of $(8.665 \times 10^3 \text{ cpm}) / (2.51 \times 10^4 \text{ dpm})$, which is equal to 0.345, or 34.5%. The theoretical geometry factor is about 33%.

There is no indication of the release of iodine upon acidification of the sample with H_2SO_4 , at least in the I^{131} concentration range studied. Neither was there any significant adsorption of I_2 on the glass walls of the reaction vessel. However, since I_2 is evolved when solutions of appreciable KI concentrations are acidified, it will still be advantageous to replace H_2SO_4 with $HC_2H_3O_2$ for the sample acidification step.

2. Hydrazine/Ammonia Analysis

The analytical procedures developed at CPP for analysis of liquid samples containing hydrazine and ammonia, have been published in IDO-14316.

Two methods will be used for hydrazine analysis, depending upon N_2H_4 concentration: An oxidimetric titration method is used for determining N_2H_4 and NH_3 in samples in which the N_2H_4 content is greater than 0.416 mg. The precision on a series of test samples containing 5 wt% N_2H_4 was better than 1% relative standard deviation for both compounds. The procedure is applicable to samples that have to be handled remotely. A colorimetric method similar to that used at AGN has been developed for samples of low N_2H_4 concentration. The lower limit of the colorimetric method is estimated as five μ gm of N_2H_4 per ml of liquid ammonia. For a series of sample aliquots containing from two to nine μ gm of N_2H_4 , an average calibration factor of 0.0882 absorbance units per μ gm of N_2H_4 was obtained. The relative standard deviation of these measurements was 0.4%. A separate aliquot is taken for NH_3 analysis by the oxidimetric titration method.

Liquid ammonia samples are reacted into 10 ml of 6M acetic acid containing 10 μ gm of iodide holdback carrier. In both of the hydrazine methods, no interference is observed from the use of either acetic acid or the iodide carrier.

During routine analysis at CPP, bench standards (within the limits specified by the Quality Control Laboratory) will be analyzed at the start of each shift. Periodically, standard samples will be submitted as normal samples by the Quality Control Laboratory. Samples for both the oxidimetric and the colorimetric methods will be analyzed over the N_2H_4 - NH_3 concentration range expected from loop samples.

3. Other Analyses

Chromatographic analysis of gas samples will require two aliquots, one for N_2 and H_2 , and another for NH_3 analysis. CPP is running mass spectrometric checks on those synthetic samples in which NH_3 was not resolved. Nitrogen-hydrogen analyses by gas chromatography appear excellent, and the method was used at CPP to check the accuracy of hydrogen monitors in the loop off-gas system.

CPP analytical personnel suggested performing the following additional periodic analyses as a periodic check on loop operations: mass spectrometric analysis of gas samples to check N_2 , H_2 , and NH_3 content;

emission spectrograph analysis of metals or solids in liquid samples; and gamma spectroscopy for I-133 and I-135 in liquid samples. Rapid transfer of samples to CPP by Health Physics truck may be required.

C. EQUIPMENT

AGN personnel will monitor activity in the gaseous effluent from the loop in the hope that, by determining a particular gaseous fission product, fission rate in the loop can be accurately predicted. Equipment for monitoring the gas stream consists of a Nuclear Data Model 101, 256-channel pulse height analyzer with a 3 x 3 in. diameter NaI(Tl) scintillation crystal. Readout is provided by an IBM typewriter and a Tektronix oscilloscope. Calibrations have been performed to determine energy per channel and zero levels at maximum energies of 3, 2, 1, and 0.3 Mev. Calibration lines for fine gain setting change versus gain change have been provided to facilitate corrections for drift. Efficiency of the detector crystal is assumed to be adequately calibrated by Heath's published efficiency curves. For high radiation levels, lead filters will be inserted between the source and the detector; 1/8-in. lead absorber calibrations have been obtained for this purpose.

VI. PROGRAM FOR NEXT PERIOD

Final testing of the revised system for manual liquid level probe operation will be completed by mid-November. A final period of ex-reactor loop operation by AGN personnel will sequentially check the validity of all instructions in the Operating Manual and serve as a final, integrated test of all equipment. Training of the PPCo operating crew in late November will be followed by loop insertion for zero power operation, starting on December 2. On December 23, following the first addition of UO_2 fuel, 1 kw operation will start, and 5 kw operation is scheduled for January 13.

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